Available online at www.sciencedirect.com

ScienceDirect

Procedia Structural Integrity 2 (2016) 1457–1464

Structural Integrity Procedia

www.elsevier.com/locate/procedia

21st European Conference on Fracture, ECF21, 20-24 June 2016, Catania, Italy

Fatigue microstructural evolution in pseudo elastic NiTi alloy

Vittorio Di Cocco[a]*, Francesco Iacoviello[a], Stefano Natali[b]

[a]*DICeM – University of Cassino and Southern Lazio, Cassino (FR) 03043, Italy*
[b]*D.I.C.M.A - "La Sapienza" University, Rome 0018, Italy*

Abstract

Shape memory property characterizes the behavior of many Ti based alloys (SMAs). This property is due to a metallurgical phenomenon, which allows to change the lattice structure without boundaries changing as a reversible transition. Equiatomic NiTi alloys are among the most industrially used SMAs: they are characterized by two different mechanical behaviors in terms of shape recovering:

- a shape memory effect (SME). This is obtained when the recovery of the initial shape takes place only after heating over a critical temperature, with a consequent crystallographic structure transition;
- a pseudoelastic effect (PE). This is obtained when the critical temperature is lower than environmental temperature. In this case, the recovery of the initial shape takes place only after unloading.

In recent years, research relating to materials of shape memory has gone in the direction of application in many fields of engineering such as aerospace or mechanical systems.
In this work the evolution of microstructural lattice has been studied taking in to account the effect of low cycles fatigue loads.

Keywords: NiTi alloy; Shape memory alloy; Ftigue

1. Introduction

Shape memory alloys (SMAs) are an interesting class of materials characterized by the ability to recover the original shape also after high values of deformation.

The ability to recover the initial shape can be classified as two different ways:

* Corresponding author. Tel.: +39.0776.2994334; fax: +39.0776.2993733
E-mail address: v.dicocco@unicas.it

10.1016/j.prostr.2016.06.185

Nomenclature

SMA	shape memory alloy
SIM	stress induced martensite
TIM	thermally induced martensite
PE	pseudo-elastic effect
NiTi	equi-atomic Nickel-Titanium alloy
XRD	X-Ray diffraction
LVDT	Linear Variable Differential Transformer
eps	engineering strain in [%]

1) the traditional shape memory effect (SME);
2) the pseudo-elastic effect (PE).

In particular, SMAs are able to recover their original shape after being mechanically deformed to a large extent, by heating up to a characteristic temperature (SME), or by removing the mechanical load (PE).

From the microstructural point of view shape memory and pseudoelastic effects are due to reversible solid state microstructural transitions from the high temperature parent austenitic phase to the low temperature product martensitic one. The phase transition can be activated by a temperature change (for example TIM) between the characteristic phase transition temperature, or by external mechanical loads as the SIM like shown by Sato et all. (1982), Liu et all. (2000) and by Di Cocco et all. (2011.).

The near equiatomic NiTi binary system shows an exploitable characteristic and it is currently used in an increasing number of applications in many fields of engineering. It is used in the realization of smart sensors and actuators, joining devices, hydraulic and pneumatic valves, release/separation systems, consumer applications and commercial gadgets. However, the most important applications of NiTi alloys are in the field of medicine, due to their good mechanical properties and biocompatibility where pseudoelasticity is mainly exploited for the realization of several components, such as cardiovascular stent, embolic protection filters, orthopedic components, orthodontic wires, micro surgical and endoscopic devices.

In this work the mechanical properties of a commercial NiTi shape memory alloy have been investigated by tensile tests of miniaturized dog bone shaped specimens carried out by using a special mini testing machine, which allows in situ XRD investigations during mechanical loading. In particular, XRD analyses have been carried out at fixed values of the applied deformation, and the direct stress induced phase transformation (SIM) has been observed during loading together with the reverse transformation after unloading.

2. Material and methods

An equiatomic NiTi alloy characterized by a PE mechanical behavior has been used in order to evaluate the structural modification in low cyclic. The equilibrium state diagram of investigated alloy is shown in Fig. 1, where a presence of a cross of limit of solution of two different phases is the peculiarity of this alloy. The influence of chemical composition is very strong in terms of mechanical behavior, because, weekly difference of Ni or Ti contents change to stability of phases and can modify the memory properties of alloy.

The thermo mechanical process carried on the investigated material, put the critical of stable austenite below than the environment temperature. As a consequence the investigated alloy is characterized by a PE behavior; it is able to recover the initial shape when loads is null also over high values of deformation.

Specimens deformation and applied load were measured by means of a Linear Variable Differential Transformer (LVDT) and two miniature load cells (10 kN each), respectively (tensile holder and the fatigue testing machine are shown in Figs 3a and 3b, respectively).

The evolution of the microstructure during uniaxial deformation was analyzed by a miniature testing machine which allows in-situ X-Ray micro-diffraction analyses.

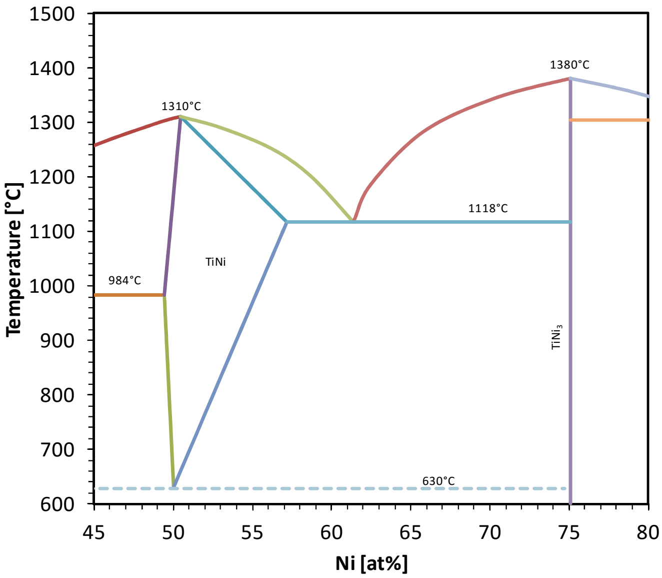

Fig. 1. NiTi alloy phase diagram.

In particular, the testing machine is equipped with a simple and removable loading frame, which allows X-Ray analyses at fixed values of applied load and/or deformations. The machine is powered by a stepping motor, which applies the mechanical deformation to the specimen through a calibrated screw, with pitch of 0.8mm, and a control electronic allows simultaneous measurement and/or control of applied load and stroke of the specimen head (Fig. 2).

The stroke is measured by a LVDT while the load is measured by two miniaturized load cells with maximum capacity of 10 kN. Miniature dog bone shaped specimens with dimension showed in Fig. 3 were machined from NiTi sheets, by wire electro discharge machining, due to the poor workability of this class of materials by conventional machining processes as well as to reduce the formation of thermo-mechanical affected zone.

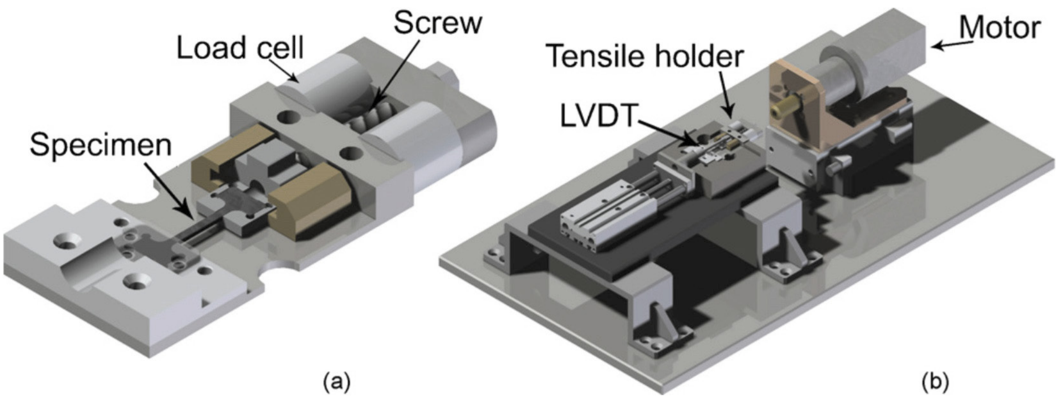

Fig. 2. Tensile holder with microtensile specimen (a); fatigue testing machine (b) (Di Cocco et all. 2011).

Step by step isothermal tensile tests were carried out, at room temperature, at increasing values of the specimen elongation. In particular, two levels of elongation have been applied:

1) Unloaded corresponding to eps=0%
2) Loaded corresponding to eps=10%

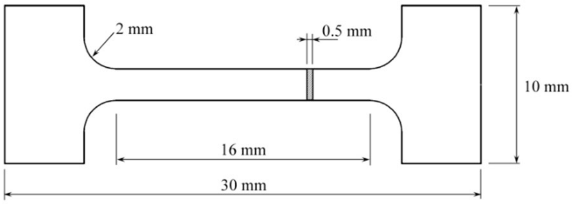

Fig. 3. Uniaxial miniaturized specimens.

For each the frame containing the specimen was removed from the testing machine, at fixed values of deformation, and analyzed by means of a Philips diffractometer in order to evaluate XRD spectra. XRD measurements were made with a Philips X-PERT diffractometer equipped with a vertical Bragg–Brentano powder goniometer.

A step–scan mode was used in the 2θ range from 40° to 95° with a step width of 0.02° and a counting time of 2 s per step. The employed radiation was monochromated CuKα (40 kV – 40 mA). The calculation of theoretical diffractograms and the generation of structure models were performed using the PowderCell software.

The XRD investigations have been performed step by step at first, at 10th, at 50th and at 100th cycle, in loading conditions. Results were compared at same conditions of imposed engineering strain, allowing to highlight the effect of cycles on the structure modifications.

3. Results and discussion

Focusing on the initial cycle (0 cycle) the initial spectrum is the typical spectrum which characterized the undeformed austenite (Fig. 4). There are three different pecks at 2θ=42.23°, 77.53° and 92.71°, respectively in order to the counts intensify; all these peaks are typical of the austenitic phases. Furthermore, the amplitude of main peak is not so high, due to good crystalline of material in the initial conditions.

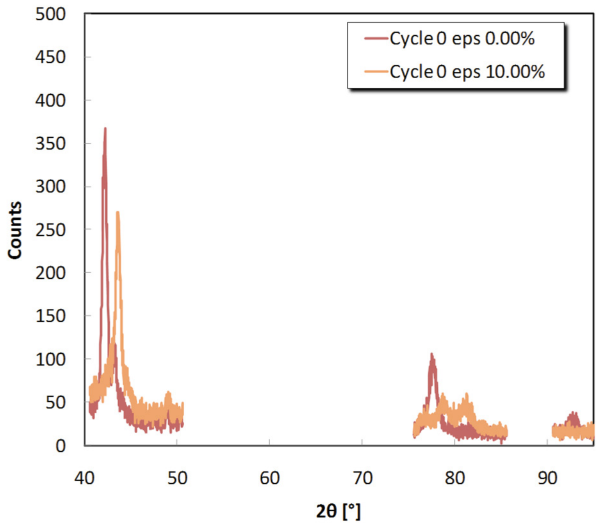

Fig. 4. Spectra of initial state (Cycle 0 and eps=0%) and of higher values of cyclic deformation (Cycle 0 and eps=10%) at first cycle.

At eps=10% (maximum value of investigated cyclic deformation) the structure of alloy is fully martensitic characterized by presence of three pecks 2θ=43.59°, 78.63° and 81.31°. This is the typical behavior of NiTi shape memory alloy, which the structure changes from a parent austenitic lattice to the martensitic lattice, transition induced by applied loads of deformations, where the stress-strain relation can be like the curve shown in Di Cocco et all. (2011).

The same specimen after 100 cycles shows both different behavior mechanical behavior different structure evolution. Focusing on the lattice structure (Fig. 5), the unloaded spectrum is typical of an austenite lattice, but at eps=10% the martensite spectrum shows the presence of a new peak at low values of angles.

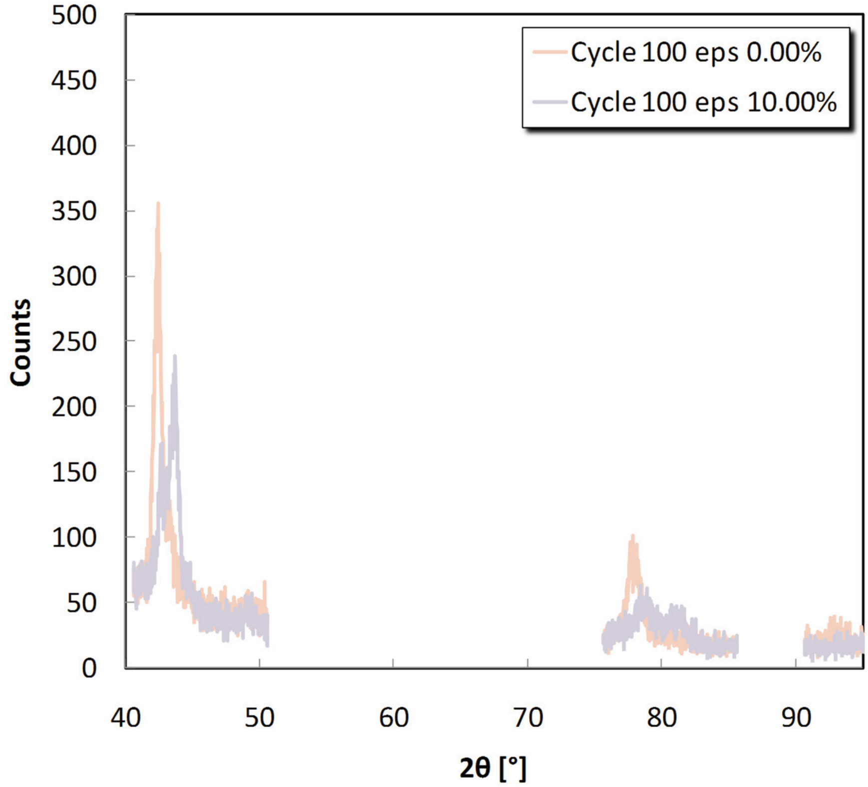

Fig. 5. Spectra of initial state (Cycle 100 and eps=0%) and of higher values of cyclic deformation (Cycle 100 and eps=10%) at last cycle.

In particular the angles which characterizes the martensite are shown in Table 1. The new peak is the second (not present in the first cycle) but its presence do not compromise the ability of SMA to recover the initial shape. In Fig. 6 is shown a comparison of austenite spectra in three different conditions, at beginning condition (not loaded and cycle 0), after first cycle in unloaded conditions and at cycle 100 also in the unloaded condition.

Table 1. An example of a table.

Peaks	Angle 2θ [°]	Relative intensity [%]
Peak 1	43.71	100
Peak 2	42.59	69
Peak 3	78.39	25
Peak 4	81.01	19

Small differences in term of angles are shown, but in terms of intensity (counts) the differences are not so negligible, due probably to different presence of defects.

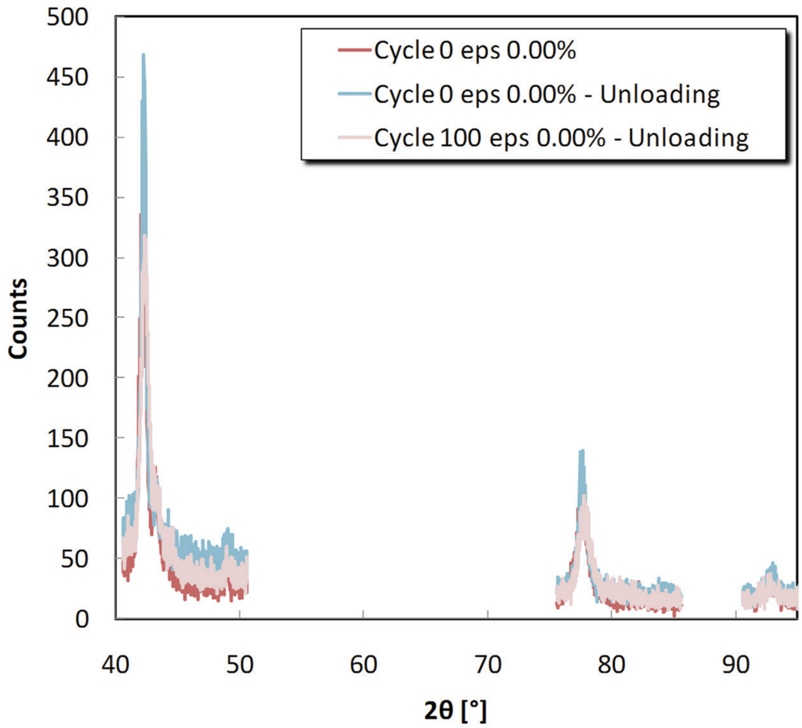

Fig. 6. Spectrum of initial state (Cycle 0 and eps=0%) compared with to the spectra of unloading condition at first and at last cycles.

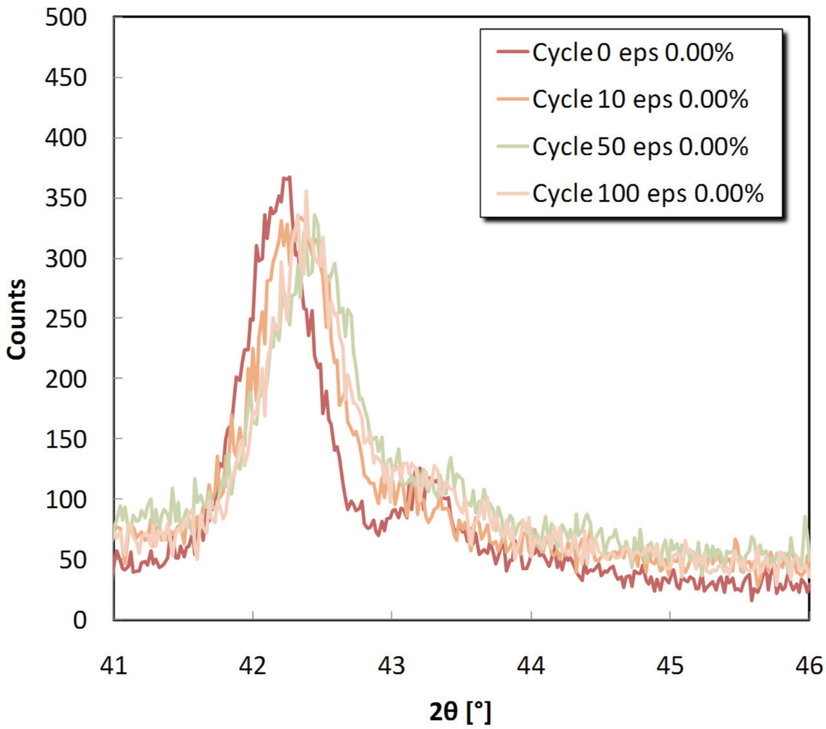

Fig. 7. Spectra of austenite at 0, 10, 50 and 100 cycles.

Focusing at low values of angles, the peaks of austenite (Fig. 7) and martensite (Fig. 9) show the influence of fatigue cycling. In terms of austenite, the angles of the main peak shift on the right: it means that the cell parameter of austenite (is a cubic cell) change for cycling effect.

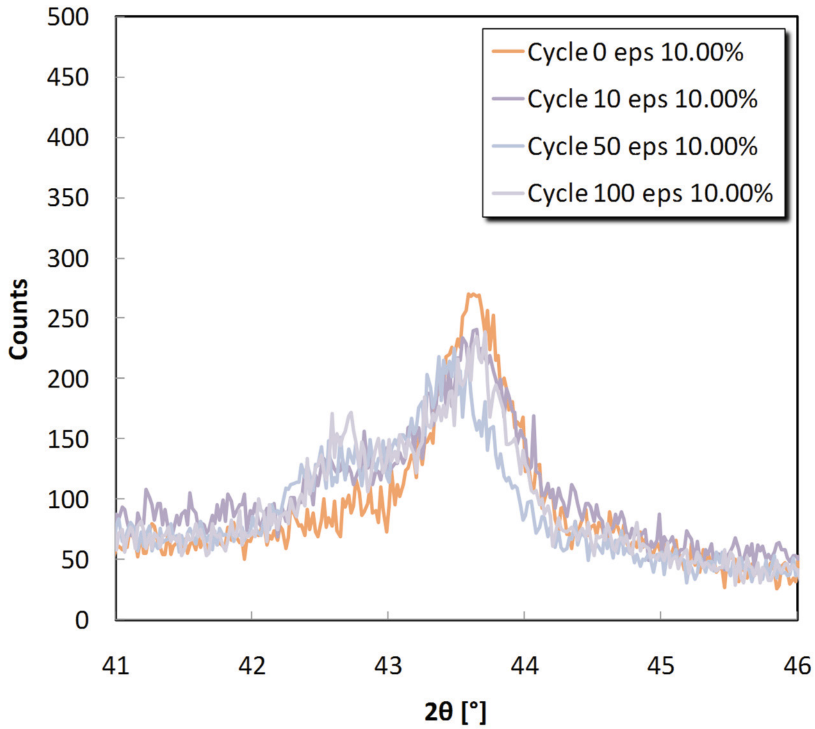

Fig. 8. Spectra of martensite at 0, 10, 50 and 100 cycles.

Analogous behavior of martensite cannot be observe (Fig. 8), because the angle of main peak is the same for each investigated cycle. However, the effect of cycling implies the presence of new peak (peak 2 in table 1 for 100 cycles), which became more and more evident at high values of cycles.

4. Conclusion

In this work a fatigue microstructural evolution of a commercial NiTi alloy, characterized by a PE behavior, has been investigated.

The analyzed diffraction spectra show the results as follows:

1) The austenite spectrum is always recovered at each investigated cycle, due to ability to recover the initial shape up to 100 cycles.
2) The austenite spectra shows a shift of the first peak on the right, as effect of fatigue cycling;
3) The effect of fatigue cycling implies a new peak of martensite at lower angles.

References

Sato, A., Chishima, E., Soma, K., Mori, T., 1982, Acta Metall, Shape memory effect in γ⇄ϵ transformation in Fe-30Mn-1Si alloy single crystals, 1177.

Liu, Y., Tan., G.S., 2000, Intermetallics, Effect of Deformation by Stress-Induced Martensitic Transformation on the Transformation Behaviour of NiTi, 8, 67-75.
Liu, Y., Favier, D., 2000, Acta Mater, Stabilisation of martensite due to shear deformation via variant reorientation in polycrystalline NiTi, 48, 3489-3499.
Di Cocco, V., Maletta, C., Natali, S., 2011, Structural transitions in a NiTi alloy: a multistage loading-unload cycle, Frattura ed Integrità Stutturale 18, 34-42.
Miyazaki, S., Kimura, 1994, Advance materials, Shape memory materials hydrides. Amsterdam: Elsevier 1101.
Otsuka, K., Ren, X. (2005) Progress in Materials Science, Physical metallurgy of Ti–Ni-based shape memory alloys, 511-678.
Dong, Y., Boming, Z., Jun, L., 2008, Materials Science and Engineering A, A changeable aerofoil actuated by shape memory alloy springs, 485, 243–250.
Russel, K.C., 1969, ASM, Phase transformation, 1219.